12 FOURIER TRANSFORM INFRARED SPECTROPHOTOMETRY (FTIR)

12.1 Introduction to Infrared Spectrophotometry

12.1.1 Objectives

Through completion of this module the trainee will develop the theoretical knowledge to be conversant in:

- The theory and applications of electromagnetic radiation;
- Properties of infrared radiation;
- The basic function and design of dispersive IR and FTIR systems;
- The theory and applications of FTIR;
- The advantages and disadvantages of both dispersive and FTIR systems; and,
- The quality assurance/quality control of the FTIR system.

12.1.2 Required Readings

12.1.2.1 FBI training course, "Infrared Spectroscopy for Trace Evidence", September 11-15, 2002.

12.1.2.2 Nicolet Corporation, "Theory of FT-IR", internal publication, 1986.

12.1.2.3 Saferstein, Richard, ed., Forensic Science Handbook, Volume 3, Englewood Cliffs, NJ, Prentice-Hall, Inc. 1993, pp.70-248.

12.1.2.4 Willard, Hobart H., Merrit, Lynne L. Jr., and Dean, John A., Instrumental Methods of Analysis, 5th edition, D. Van Nostrand Co., New York, New York, 1974, pp. 150-188.

12.1.3 Questions

The trainee will provide written answers to the following questions:

- Describe the electromagnetic spectrum.
- What is infrared spectrophotometry and what is its specificity?
- Define the following terms:
  - Wavelength
  - Frequency
  - Dipole moment
  - Harmonic vibration
  - Fundamental vibration
  - Interferometer
  - Overtones
  - Data spacing
  - Interferogram
  - Zero path difference (ZPD)

- What are the upper and lower limits of the infrared region of the electromagnetic spectrum?
- What region is the most useful analytically?
- What two conditions must be present for infrared absorption to occur?
- What is the intensity of an infrared absorption proportional to?
- What is meant by vibrational coupling?
- Describe the different types of detectors available for infrared instruments.
- What is spectral subtraction and how is it useful?

- Describe how an FTIR instrument works.
- What is the relationship between resolution and data spacing?
- Describe reflectance analysis using the microscope attachment.
- Draw a schematic diagram for the dispersive IR and the FTIR.
- What are the advantages of FTIR over dispersive instruments?
- Describe the QC procedures and preventative maintenance schedule performed on the FTIR.

12.1.4 Practical Exercise

12.1.4.1 The trainer will demonstrate the daily and weekly QC procedures for the bench.

12.1.4.2 The trainee will perform the daily and weekly QC procedures for the bench for a minimum of one week.

12.1.5 Evaluation

12.1.5.1 The trainer will review the written answers to the questions with the trainee.

12.1.5.2 The trainer and the trainee will review and discuss the pertinent points of each of the required readings.

12.1.5.3 Review of practical exercise.

12.1.5.3 The trainee will be quizzed orally upon the subject matter.

12.2 Sample Preparation and Data Collection

12.2.1 Objectives

Through completion of this module the trainee will have developed and demonstrated theoretical knowledge and/or practical skills to:

- Prepare samples and collect infrared data with the bench using the following sample preparation techniques:

  Solids:
  KBr pellet
  Diffuse reflectance
  Attenuated Total Reflectance (ATR) – as available
  Liquids:
  Film on KBr pellet
  Diffuse reflectance
  Gases:
  Gas cell

12.2.2 Required Readings

12.2.2.1 FBI training course, “Infrared Spectroscopy for Trace Evidence”, September 11-15, 2002.

12.2.2.2 Saferstein, Richard, ed., Forensic Science Handbook, Volume 3, Englewood Cliffs, NJ, Prentice-Hall, Inc. 1993, pp. 70-248.

12.2.2.3 Miller, R.G.J., Laboratory Methods in Infrared Spectroscopy, Heyden and Sons Ltd., 1965.

12.2.3 Questions

The trainee will provide written answers to the following questions:

- Why are alkali halides used for sample holders?
- What is the difference between diffuse reflectance and attenuated total reflectance?
- What parameters can be changed to improve the quality of a spectra?
- What is the background and why is it collected?

12.2.4 Practical Exercises

5.2.4.1 The trainer will demonstrate any of the sample preparation techniques with which the trainee is not familiar.

5.2.4.2 Using samples provided by the trainer, the trainee will demonstrate the ability to prepare samples using the listed sample preparation techniques.

12.2.5 Evaluation

12.2.5.1 The trainer will review the written answers to the questions with the trainee.

12.2.5.2 The trainer and the trainee will review and discuss the pertinent points of each of the required readings.

12.2.5.3 Review of practical exercises.

12.3 Infrared Interpretation

12.3.1 Objectives

Through completion of this module the trainee will have developed and demonstrated theoretical knowledge and/or practical skills to:

- Interpret FTIR data;
- Compare date collected with reference samples for identification; and,
- Compare data collected from known and questioned samples to determine whether they may or may not be associated.

12.3.2 Required Readings

12.3.2.1 Bellamy, L. J., The Infrared Spectra of Complex Molecules, John Wiley and Sons, New York, 1954.

12.3.2.2 Cook, B.W. and Jones, K., A Programmed Introduction to Infrared Spectroscopy, Heyden and Sons Ltd., 1972.

12.3.2.3 Syzmznski, Herman A., Interpreted Infrared Spectra, Plenum Press Data Division, New York, 1967.

12.3.3 Questions

The trainee will provide written answers to the following questions:

- State the absorption region for the following functional groups
  - O-H
  - N-H
  - C=O

- C-O
- $C-H_n$
- C≡N
- $N-O_2$

- What is the minimum percent of a compound needed for detection by FTIR?

12.3.4 Practical Exercises

12.3.4.1 The trainee will interpret spectra provided by the trainer.

5.3.5 Evaluation

12.3.5.1 The trainer will review the written answers to the questions with the trainee.

12.3.5.2 The trainer and the trainee will review and discuss the pertinent points of each of the required readings.

12.3.5.3 Review of practical exercises.

12.4 FT-IR Spectra Tech IR Plan Microscope Accessory

12.4.1 Objectives

Through completion of this module the trainee will have developed and demonstrated theoretical knowledge and/or practical skills to:

- Align the microscope and perform the weekly QC; and,
- Prepare samples and collect infrared data with the microscope using the microcompression cell with diamond windows.

12.4.2 Required Readings

12.4.2.1 Reffner, John A. and Martoglio, Pamela A., "Uniting Microscopy and Spectroscopy" in Practical Guide to Infrared Microspectroscopy, Humecki, Howard J., ed., Marcel Dekker, Inc., New York, pp. 41-84.

12.4.2.2 Saferstein, Richard, ed., Forensic Science Handbook, Volume 3, Englewood Cliffs, NJ, Prentice-Hall, Inc. 1993, pp. 70-248.

12.4.3 Questions

The trainee will provide written answers to the following questions:

- Why is the MCT detector cooled with liquid nitrogen?
- What is the benefit of using the MCT detector with the microscope attachment and not the DTGS detector?
- What is the range of an MCT detector and what is the limiting factor which dictates how low it will detect?
- What are interference fringes? Why do they occur? How can they be avoided?
- How does the amount of pressure applied effect samples in the microcompression cell?
- KBr is always added with samples when using the microcompression cell. Why?

12.4.4 Practical Exercises

12.4.4.1 The trainer will demonstrate the daily and weekly QC procedures for the microscope.

12.4.4.2 The trainee will perform the weekly QC procedures for the microscope for a minimum of one month.

12.4.4.3 The trainer will demonstrate sample preparation using the microcompression cell if the trainee is not familiar with this technique.

12.2.4.2 Using samples provided by the trainer, the trainee will demonstrate the ability to prepare and analyze samples using the microcompression cell.

12.5 Evaluation

12.5.1 The trainer will review the written answers to the questions with the trainee.

12.5.2 The trainer and the trainee will review and discuss the pertinent points of each of the required readings.

12.5.3 Review of practical exercises.

12.6 Competency Evaluation and Mock Trial

The trainee will use FTIR when completing their subdiscipline competency test and will defend their results as a part of their mock trial in that subdiscipline.

12.7 Reading List

12.7 1 Advanced Microspectroscopic Solutions Seminar, Spectra Tech.

12.7.2 Bellamy, L. J., The Infrared Spectra of Complex Molecules, John Wiley and Sons, New York, 1954.

12.7.3 Cook, B.W. and Jones, K., A Programmed Introduction to Infrared Spectroscopy, Heyden and Sons Ltd., 1972.

12.7.4 FBI training course, "Infrared Spectroscopy for Trace Evidence", September 11-15, 2002.

12.7.5 Humecki, Howard J., Ed., Practical Guide to Infrared Microspectroscopy, Westmont, Illinois, McCrone Associates, 1985.

12.7.6 Miller, R.G.J., Laboratory Methods in Infrared Spectroscopy, Heyden and Sons Ltd., 1965.

12.7.7 Nicolet Corporation, "Theory of FT-IR", internal publication, 1986.

12.7.8 Saferstein, Richard, ed., Forensic Science Handbook, Volume 3, Englewood Cliffs, NJ, Prentice-Hall, Inc. 1993.

12.7.9 Syzmznski, Herman A., Interpreted Infrared Spectra, Plenum Press Data Division, New York, 1967.

12.7.10 Willard, Hobart H., Merrit, Lynne L. Jr., and Dean, John A., Instrumental Methods of Analysis, 5th edition, D. Van Nostrand Co., New York, New York, 1974.

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